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STATE UNIV OF NEW YORK AT BUFFALO DEPT OF CHEMISTRY  
ELECTROCHEMISTRY AND ELECTROCHEMICAL METHODOLOGY IN MOLTEN SALT--ETC(U)  
SEP 80 R A OSTERYOUNG, J O OSTERYOUNG  
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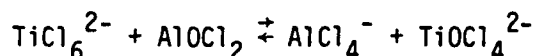
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takes place to lead to the formation of viologen species.

The deposition of Al from this melt and the melt mixed with benzene as a cosolvent was studied; it was found that the acid-base properties of the solvent are unchanged when diluted with benzene.

The oxidation of a number of aromatic amines - triphenylamine, N,N-dimethylaniline, N,N-dimethyl-p-toluidine, diphenylamine and N,N'-tetramethylbenzidine - was investigated in the melt. In the simplest case, that of triphenylamine, reversible oxidation to a radical cation was found which undergoes further oxidation, presumably to an unstable dication. The stability of the radical cation varies with melt acidity, being most stable in the acid (excess  $\text{AlCl}_3$ ) melts. The behavior of the other amines is more complicated.

The electrochemistry of Ti(IV) in a basic melt was investigated; it was found that two Ti(IV) species can exist in slow equilibrium in the melt. One species is a chlorocomplex, the other an oxy-chlorocomplex. The reaction appears to be:



with an equilibrium constant of about 900 at 40°C. The reduction of both species appears to yield  $\text{TiCl}_6^{3-}$ .

Double layer capacity measurements were made at a Hg electrode in these molten salts; the useful range of the electrode is much less than for glassy carbon, and evidence of strong  $\text{Cl}^-$  adsorption is noted.

A series of iron-diimine complexes were studied in another room temperature molten salt (a 2:1 mole ratio mixture of aluminum chloride and ethyl pyridinium bromide). Correlations between half-wave potentials and various substituent parameters are presented.

Use of the infra-red window of these melts has permitted correlation of the electrochemical behavior of anthraquinone in the melt to observed infra-red spectral changes associated with carbonyl-aluminum chloride interaction.

Aspects of techniques in development for studying electrode processes at solid electrodes are discussed.

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**AFOSR-TR- 80 - 1371**

This is a Final Report on <sup>F49620-79-C-0117</sup> [REDACTED], "Electrochemistry and Electrochemical Methodology in Molten Salts."

The period of the contract was from 1 June 1979 to 30 September 1980, and the contractual amount was \$130,315.

## SUMMARY OF WORK SINCE 1 JUNE, 1979

### I. Move to New York

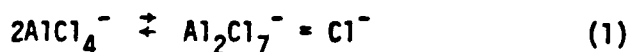
In mid June, 1979, the principal investigators moved from Colorado State University, Ft. Collins, Co., to the State University of New York at Buffalo. Research work at Colorado was terminated in late May, and the move, which included almost all equipment and five post docs, was performed in early June. Portions of the move were performed by commercial movers. The move went surprisingly smoothly in most respects; we anticipated problems with our dry box systems, but two of the three boxes were actually operative, and experiments being performed, two weeks after arrival. It has taken a good deal longer to get our total laboratory together, particularly one of the computers we moved, an ancient Digital Equipment PDP-12 (~11 years old), partially purchased on AFOSR funds. However, it too is finally operative. Our DEC PDP 8/e, in Dr. Janet Osteryoung's lab, a much newer system, was operative for on-line work very shortly after arrival in New York. We have greatly increased the amount of laboratory space available to us, and feel, overall, that the move went much more smoothly than we anticipated.

### II. Summary of Research in Chloroaluminate Melts

Most of the work carried out during this period involved studies in the "room temperature" melt, N-butylpyridinium chloride-aluminum chloride. (BuPyCl:AlCl<sub>3</sub>.) This melt, with a liquidus at the 1:1 mole ratio of ~ 27°C, was discovered in our laboratory about 3 and 1/2 years ago and has engendered considerable interest by others in efforts

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to both utilize it and find similar, low melting molten salt systems (26,28,30). The dominant equilibrium



was determined as  $\leq 3.8 \times 10^{-13}$  and the mole ratio of  $\text{BuPyCl}:\text{AlCl}_3$  can be varied from about 1:0.65 to 1:2 - i.e., from very basic (excess  $\text{BuPyCl}$ ) to very acidic (excess  $\text{AlCl}_3$ ). As reported previously, the 1:1 melt is essentially  $\text{BuPy}^+ \text{AlCl}_4^-$ , while the 1:2 melt is essentially  $\text{BuPy}^+ \text{Al}_2\text{Cl}_7^-$  (26). Spectroscopic work, supported in part by the Office of Naval Research, has been used to establish some of the fundamental solvent characteristics.

#### 1) Reduction of Butylpyridinium cation

In determining the solvent equilibria, it was noted that  $\text{Al}$ , in the basic melt, reacted with the melt to form a brightly colored entity.

As a result, the reduction of 1-alkylpyridinium cation in 1:0.8 mole ratio N-butylpyridinium chloride:  $\text{AlCl}_3$  at  $40^\circ\text{C}$  was investigated by cyclic voltammetry and electrochemical pulse methods. A dimeric product of the primary pyridinyl radical coupling, 4,4'-tetrahydrobipyridine, forms in about 45% yield and undergoes fairly rapid chemical dissociation to the stable, electroactive, 1,1'-dibutyl-4,4'-bipyridinium monocation radical. The corresponding biquaternary bipyridyl base reduces in two steps, each with one-electron reversible behavior, at half-wave potentials of  $-0.56 \text{ V}$  and  $-0.96 \text{ V}$  vs.  $\text{Al}(0)$  in 1:2  $\text{BuPyCl}:\text{AlCl}_3$  reference electrode. Tetrahydrobipyridine species with 1,2,4,6-substituted methyl groups were stable to dissociation under similar conditions.

A manuscript based on this work has been published in the Journal of the Electrochemical Society (34).

## 2) Melt and co-solvent

As reported in the Final Report in AFOSR-76-2978, we have studied the behavior of  $\text{AlCl}_3$ -BuPyCl in mixtures with benzene, and found that benzene functions, essentially, as a diluent without significantly altering the acid-base properties of the solvent or the electrochemistry of solutes. The paper based on this work has now been published in J. Electrochem. Soc. (32).

## 3) NMR studies of acidity

One of our interests has long been the relative strength of the acid species in the melt. Work initiated at Colorado State, and continued at SUNY/AB, involved NMR studies of the  $^{13}\text{C}$  shifts on the pyridinium ring as a function of melt acidity. A plot of the  $^{13}\text{C}$   $\text{C}_2$ - $\text{C}_4$  shifts by the butylpyridinium cation in a variety of solvents of known Hammett acidity is linear and has been compared to that for the  $\text{BuPy}^+$  in the BuPyCl- $\text{AlCl}_3$  melts at various mole ratios of BuPyCl: $\text{AlCl}_3$ . (In the latter case, the shifts were measured and placed on the 1M  $\text{BuPy}^+$  line.)

While very qualitative in nature, the data indicate the strongly Lewis acid - but widely variable-nature of the  $\text{AlCl}_3$ :BuPyCl melts. Additional work related to this has been performed here, and we anticipate preparing a manuscript for J. Am. Chem. Soc.

## 4) Oxidation of Aromatic Amines

In keeping with our efforts to compare and contrast these melts with the  $\text{NaAlCl}_4$  system a study of the electrochemical oxidation of a number of aromatic amines was made.

The electrochemical oxidation of a number of aromatic amines (triphenylamine (TPA), N,N-dimethylaniline (DMA), N,N-dimethyl-p-toluidine (DMT); diphenylamine (DPA and N,N'-tetramethylbenzidine (TMB)) in the low temperature molten salt system BuPyCl:AlCl<sub>3</sub> has been investigated. In acidic melts TPA is oxidized reversibly to its radical cation which at more positive potentials undergoes a further, chemically irreversible oxidation to the dication of the amine. In basic melts the stability of the radical cation is reduced, and it couples to form tetraphenylbenzidine which is itself further oxidized. The oxidation of the other amines was more complex as a result of their being involved in complexation equilibria with acid species in the melt. In basic melts where they were uncomplexed their behavior was similar to that of TPA but in acidic melts the amine complexes underwent irreversible two electron oxidations to their respective dications at potentials approximately one volt positive of the first oxidation potential of the free amine. TMB which, depending on the melt acidity, could be present as the free amine, mono or di-complex was also oxidized to its dication. The actual mechanism of this oxidation was dependent upon the melt acidity. The oxidation of TMB was also shown to be dependent upon the temperature, the TMB cation radical being stable at low temperature but becoming unstable with respect to disproportionation as the temperature was increased.

This work has been published in J. Am. Chem. Soc. (33).

#### 5) Electrochemistry of Ti and Oxide

We have carried out some work on the electrochemistry of Ti(IV) in BuPyCl:AlCl<sub>3</sub> which has recently been completed.



We found that Ti(IV) is reduced to Ti(III) in a reversible one-electron reduction in the basic BuPy1:AlCl<sub>3</sub> melt. TiCl<sub>4</sub> is readily introduced as the liquid into the melt. The half wave potential for the reduction of Ti(IV) to Ti(III) is -0.322 V vs. Al(0) in the 1:2 BuPy1:AlCl<sub>3</sub> melt. A second wave appearing at more negative potentials has been subjected to a great deal of study; initial experiments indicated that the ratios of the wave heights in the apparent two-step reduction of Ti(IV) varied in a random manner. A good deal of effort was expended in melt purification, and it has been demonstrated conclusively that the second wave is the result of the reduction of a Ti-O complex, formed as a result of oxide impurity in the melt. Under all conditions, only one reoxidation wave is observed for the reverse scan (oxidation of Ti(III)), and we have shown that a reaction



takes place, for which the equilibrium is slow; the first wave is the reduction of  $\text{TiCl}_6^{2-}$ , while the second is the reduction of  $\text{TiOCl}_4^{2-}$ , with a difference in reduction potential of about 0.4 volts between the two Ti(IV) species. The equilibrium constant for Eq. 1 is 900 at 40°C.

Coulometric experiments indicate that all the Ti(IV) is reduced at potentials negative of either the first or second wave ( $n=1$ ), verifying slow equilibrium between two Ti(IV) species. The addition of oxide (as Li<sub>2</sub>CO<sub>3</sub>) decreases the height of the first Ti(IV) reduction wave and increases the height of the second. A manuscript based on this work is in preparation, and it appears that the reaction can be used to determine oxide content in these melts (38).

#### 6) Use of Hg electrodes

A brief study of the use of an Hg electrode in the melt has been carried out. Double layer capacity measurements have been made. The useful potential range is drastically decreased over that at glassy carbon electrodes and evidence for chloride adsorption is presented. A paper on this work has been published in *Electrochimica Acta* (36).

#### 7) New melts

A limited effort has gone into the preparation of new melts. Within the past few months we have made a melt of p-dimethylamino-N-butylpyridinium chloride-aluminum chloride. Preliminary measurements indicate a cathodic limit ca. -0.6 V more negative at glassy carbon in the basic melt, compared to BuPyCl-AlCl<sub>3</sub>; no "colored" species form at the cathodic limit, but it is not clear that Al is being deposited. The acid-base equilibrium constant has been determined.

#### 8) I<sup>-</sup>/I<sub>2</sub> Redox Couple

Studies on the behavior of I<sup>-</sup>/I<sub>2</sub> redox couple in the melt have been undertaken. The work is not complete, but the chemistry is much more complicated than anticipated, showing evidence of fast follow-up redox reactions.

#### 9) Iron Diimine Complexes

A piece of work carried out several years ago at Colorado State University under AFOSR support has now been written up and submitted for publication. It is a continuation of work (20) reported under the Final Report to AFOSR 76-2978. An abstract of a paper, submitted to *Inorganic Chemistry* (40), follows:

"The study of thermodynamic and spectral properties of the iron diimine complexes is extended to a solvent system based on molten aluminum chloride and ethyl pyridinium bromide in a 2:1 molar ratio. This solvent system presents an important advantage over acetonitrile being a totally anhydrous medium, in which ligand-oxidation processes of these complexes, found to occur in aqueous sulfuric acid and to a smaller extent in acetonitrile, do not take place. The effect of the substituents on the iron diimine chromophore on the half-wave potentials,  $E_{1/2}$ , of the  $\text{FeL}_3^{3+}/\text{FeL}_3^{2+}$  couples and on the frequencies of the inverse charge transfer band is compared to that exhibited in other media. In this molten salt the thermodynamic stability of the ferric forms is greater than that of the ferrous forms, whereas the opposite trend exists in aqueous solutions. Correlations between  $E_{1/2}$ 's and Taft's polar and steric parameters of the substituents are presented and compared to those encountered in other solvents."

#### 10) Spectroscopic Studies

While not funded by AFOSR, work involving spectroscopic studies to correlate electrochemical behavior to solute electrochemistry in the room temperature molten salt systems is related to the AFOSR electrochemical studies, since we have clearly made use of information garnered here to further the spectroscopic work, which is supported by the Office of Naval Research. Simply for the sake of completion, and because in some instances the support of both agencies will be and has been acknowledged on publications, that work is summarized here.

We have found that these room temperature molten salts possess a reasonably useful infra-red window. A paper on our first results has been published in Inorganic Chemistry (37) and its abstract follows:

A simple, direct transmission infrared technique has been applied to obtain spectra of acidic and basic aluminum chloride-1-butylpyridinium chloride melts. Band assignments for the  $\text{AlCl}_4^-$  ion show splitting of the  $\nu_3$  mode which confirms that tetrahedral distortion occurs in this melt environment. Comparison of the bands for the  $\text{Al}_2\text{Cl}_7^-$  ion in solid  $\text{Te}_4(\text{Al}_2\text{Cl}_7)_2$  and the anion species in the 2:1 mole ratio melt, in the  $200\text{-}650\text{-cm}^{-1}$  region, supports the premise that the ion in the liquid phase has a bent Al-Cl-Al bridge. Detailed spectra also have been obtained of the 1-butylpyridinium cation, and the absence of extensive band broadening may indicate that the melt structure is quasi-crystalline.

Following this, we have studied in some detail the chemistry and electrochemistry of anthraquinone in these melts, making use of the infra-red window to study and correlate the infra-red and electrochemical behavior. A manuscript on this work has been submitted to J. Am. Chem. Soc., and has been reviewed, but it has been suggested that the material is more appropriate for J. Electrochem. Soc.; following some revision, it will be submitted to that journal (41). An abstract of that paper follows:

The electrochemical and spectroscopic properties of 9,10-anthraquinone (AQ) in the low-temperature  $\text{AlCl}_3$ :n-butylpyridinium chloride (BuPyCl) molten salt system have been studied as a function of melt acidity. Infrared spectroscopic data indicate that AQ exists in the

uncomplexed state in the basic melt (0.8  $\text{AlCl}_3$ :1.0 BuPyCl). The electrochemical behavior in this region involves a single-process two-electron reduction (with slow electron transfer) of AQ to its dianion, the reduction mechanism probably proceeding by an ECE pathway. The complexation of AQ by  $\text{Al}_2\text{Cl}_7^-$  in the acidic melt (1.2  $\text{AlCl}_3$ :1.0 BuPyCl) produces  $\text{AQ}\cdot 2\text{AlCl}_3$  as indicated both by infrared spectroscopy and chemical analysis. This complexation results in a shift in potential for the reduction process compared to the corresponding potential for AQ reduction in the basic melt of +1.4V. The reduction of  $\text{AQ}\cdot 2\text{AlCl}_3$  also involves a single-wave two-electron process. Since the same separation in potentials for  $\text{AQ}\cdot 2\text{AlCl}_3$  reduction and subsequent oxidation in the acidic melt was observed as that seen for AQ in the basic melt, some interaction of the dianion with the acidic melt is also evident. As the composition of the melt was varied through the neutral region (approx. 1.0  $\text{AlCl}_3$ :1.0 BuPyCl), in which the acidity is changing rapidly, an additional process due to reduction of  $\text{AQ}\cdot \text{AlCl}_3$  was observed; by adjustment of melt acidity by small additions of  $\text{AlCl}_3$  or BuPyCl, a melt containing all three species AQ,  $\text{AQ}\cdot \text{AlCl}_3$ , and  $\text{AQ}\cdot 2\text{AlCl}_3$  could be obtained. Electrochemical studies of this system indicated that interconversion among the various species upon reduction is rather slow.

#### 11) Semi conductor Electrode Studies

Again, the work is not supported by AFOSR, but does involve studies in the room temperature molten salt systems. In collaboration with Dr. Arthur Nozik and Dr. John Turner at the Solar Energy Research Institute and with some funds for equipment from the Office of Naval

Research, we are initiating studies on semiconductor electrochemistry in the room temperature molten salts. At present, we have evaluated some equipment for this work and are in the process of ordering equipment to permit the work to get underway.

#### 12) Laser Materials in the Room Temperature Molten Salts

Collaborative work involving Professor Mark Sceats at the University of Rochester has been underway for some time following a visit by Professor Sceats to Buffalo last winter. We had found that rare-earths, such as neodymium, are quite soluble in these melts. Professor Sceats has measured Nd(III) excited state lifetimes at the 1.06 micron lasing transition, and found them to be several microseconds. He has a student attempting to observe lasing action of the Nd(III) in several melt samples we have supplied. Professor Sceats believes there is no reason why the material will not lase, but has experienced difficulty in putting the laser cavity together. The melts, however, appear to be quite stable photochemically, a prime requirement if they are to be used effectively in a liquid laser system.

13) As a result of a visit by Professor Barry Welch last Fall, a review article on some of the work was prepared and presented by Professor Welch at the Fifth Australian Electrochemistry Conference in Perth during August, 1980. The material will be published in J. Electroanal. Chem. (39). A review previously written has been received in galley form and should appear in the near future (35).

### III. Technique Development

In the area of technique development our efforts fall into two separate categories. The first involves developing a flexible capability to do pulse voltammetric experiments under computer control; the second concerns development of pulse techniques especially appropriate for use in the melts. These will be described in turn.

#### A. Computer-controlled system for pulse voltammetry in the melts.

The application of pulse voltammetry to molten salts presents formidable problems. Because of the many-faceted nature of the work it is necessary to be able to employ a variety of techniques, and in particular, to employ techniques not available through commercial instrumentation. This requires either specialized analog circuitry or a computer-controlled system in which changes in technique can be made under software control. We have chosen the latter option, which has advantages also in universality of application and in data handling. There are two important problems which present themselves with this approach. First, our mode of experimentation requires that the equipment be brought to the experiment, rather than vice versa, and second, the users are in general naive about computers and cannot afford the large amounts of time required to become expert.

Our approach to the first problem is to produce an experimental station at each drybox which includes all of the necessary analog electronics and is directly connected to a computer. This effort is now underway. One experimental station has been constructed and tested extensively and is in the process of being reproduced.

The approach to the second problem is to develop computer software

which is sufficiently well-documented and transparent to the user that it can be used reliably by non-experts. An example of material displayed to the user and the user response is shown in Table I. The objective of this approach is to keep the focus of the work firmly on molten salt chemistry and electrochemistry rather than on computer applications. Dr. John O'Dea has been responsible for this portion of the work.

B. Development of pulse voltammetric techniques for use in the melts.

There are two main lines of work in technique development. Each of them is related to other work being carried out under NSF support. These will be described in turn.

1. In collaboration with Emilia Kirowa-Eisner of Tel-Aviv University, we have been developing the technique of reverse pulse (RP) voltammetry for the study of coupled chemical reactions at solid electrodes. One paper on this technique has appeared in Anal. Chem. (42). A second paper on coupled chemical reactions is in press in J. Phys. Chem. These papers acknowledge NSF support (43).

Another aspect of reverse pulse voltammetry arises out of our efforts to obtain correction factors for the application of RP voltammetry at spherical electrodes. This has given rise to a theoretical analysis of edge effects which provides the basis for the accurate determination of diffusion coefficients. This is especially important for determining the diffusion coefficients of unstable species. This work will be submitted to J. Electroanal. Chem. (45).



2. The second technique being developed for use in the melts is differential normal pulse (DNP) voltammetry. The wave form for this technique is shown in Figure 1. The current output is the current at  $E_2$  and  $t_2$  minus the current at  $E_2'$  and  $t_2$  (see Figure 1). This technique is being developed based on related work supported by NSF (46,47). The reason for developing this waveform is to obtain the best features of both normal pulse and differential pulse voltammetry. This is especially important in the case of glassy carbon electrodes, for the electrode in this technique can be held at a potential which minimizes film formation. At the same time, one obtains readily analyzable peak-shaped current-potential response.

A case of special interest is the totally irreversible electron transfer. We have examined this case both theoretically and experimentally using the oxidation of Fe(II) in aqueous sulfuric acid medium (48,49). The most noteworthy result is the substantial enhancement of the current for irreversible reactions over that which would be obtained by conventional techniques such as differential pulse voltammetry. This result is both predicted theoretically and verified experimentally.

#### IV. Interaction with AirForce and other DOD Laboratories

Since leaving Colorado State University, no formal visits have been made to Air Force Laboratories; discussions have been held at various meetings with individuals from both Air Force and Navy laboratories. Dr. John Wilkes of the Frank J. Seiler Research Laboratory is expected to spend two weeks here early in December.

V. Grant Personnel

Individuals associated with this research activity for any prolonged period time during the period 1 June, 1979 to 30 September, 1980 are listed below. All were at the post-doctoral level or a Visiting Scientist.

Dr. Graham Cheek

Dr. Hogne Linga

Dr. Barry Welch

Dr. John O'Dea

Dr. Emilia Kirowa-Eisner

TABLE I.

CHANGE PARAMETERS ? (Y OR N) <u>Y</u>			
NUMBER	PARAMETER	CURRENT VALUE	LIMITS
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3	POTENTIOSTAT OFFSET	0.0000	VOLTS
4	POTENTIOSTAT RANGE	-1.0	VOLTS
5	POTENTIOSTAT GAIN	3	
6	INITIAL AND FINAL POTENTIALS	-0.1000, -0.9000	VOLTS
7	STEP HEIGHT, AMPLITUDE	5.0000, 25.0000	MV
8	SQUARE WAVE FREQUENCY	400.00	HZ
9	DELAY TIME BEFORE SCAN	0.30	SEC
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SAVE DATA ? (Y OR N) <u>N</u>			
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CHANGE PARAMETERS ? (Y OR N) <u>N</u>			
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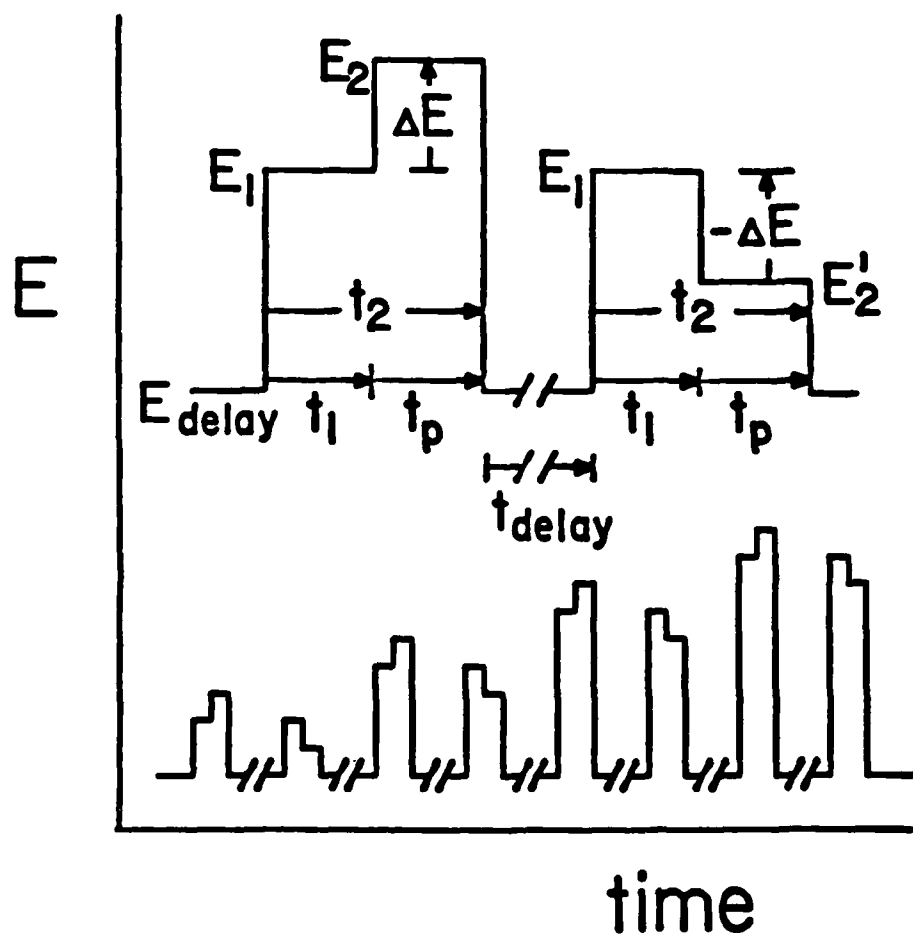


Figure 1

## APPENDIX I

### Publications--Grant Related Activity - since AFOSR support initiated.

AFOSR-71-1995; 1 Jan. 1971 - 28 Feb. 1975; Final Report 30 June 1975.

1. Janet Osteryoung and R.A. Osteryoung, "The Advantage of Charge Measurements for Determining Kinetic Parameters," *Electrochimica Acta*, 16, 525 (1971).
2. R.A. Osteryoung, "Introduction to the On-Line Use of Computers in Electrochemistry," Vol. II, "Application of Computers to Chemical Instrumentation," Ed. by Mattson, Mark and MacDonald, Marcel Dekker (1973).
3. L.G. Boxall, H.L. Jones and R.A. Osteryoung, "Solvent Equilibria in  $\text{AlCl}_3$ -NaCl Melts," *J. Electrochem. Soc.*, 120 (2), 223 (1973).
4. H. Lloyd Jones, L.G. Boxall and R.A. Osteryoung, "Organic Electrode Reactions in Fused  $\text{AlCl}_3$  Containing Solvents," *J. Electroanal. Chem.*, 38, 476 (1972).
5. L.G. Boxall, H.L. Jones and R.A. Osteryoung, "Electrochemical Studies on Ag, Fe and Cu Species in  $\text{AlCl}_3$ -NaCl Melts," *J. Electroanal. Chem.*, 121, 212 (1974).
6. H. Lloyd Jones and R.A. Osteryoung, "Electrode Reactions of Aromatic Amines in Solvents Containing Fused  $\text{AlCl}_3$ , II.", *J. Electroanal. Chem.*, 49, 281 (1974).
7. R.J. Gale and R.A. Osteryoung, "Investigation of Subvalent Ion Effects During Aluminum Anodization in Molten NaCl- $\text{AlCl}_3$  Solvents," *J. Electrochem. Soc.*, 121, 983 (1974).
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- (32) The Electrochemical Behavior of Aluminum in the Low Temperature Molten Salt System n-Butyl Pyridinium Chloride; Aluminum Chloride and Mixtures of this Molten Salt with Benzene, J. Robinson and R. A. Osteryoung, *J. Electrochem. Soc.*, 127, 122 (1980).
- (33) An investigation into the Electrochemical Oxidation of Some Aromatic Amines in the Room Temperature Molten Salt System  $AlCl_3$ :N-Butylpyridinium Chloride, J. Robinson and R. A. Osteryoung, *J. Am. Chem. Soc.*, 102, 4415 (1980).
- (34) Electrochemical Reduction of Pyridinium Ions in Ionic Aluminum Chloride-Alkylpyridinium Halide Ambient Temperature Liquids, R. J. Gale and R. A. Osteryoung, *J. Electrochem. Soc.*, 127, 2167 (1980).
- (35) H. L. Chum, and R. A. Osteryoung, "Chemical and Electrochemical Studies in Room Temperature Aluminum Halide Containing Melts," in *Ionic Liquids* ed. by D. Inman and D. Lovering, Plenum Press, in press.
- (36) Electrical Double layer at Mercury in Room Temperature Aluminum Chloride:1-Butylpyridinium Chloride Ionic Liquids, R. J. Gale and R. A. Osteryoung, *Electrochimica Acta*, 25, 1527 (1980).

- (37) Infrared Spectral Investigations of Ambient Molten Aluminum Chloride:1-Butylpyridinium Chloride Systems, R. J. Gale and R. A. Osteryoung, *Inorg. Chem.*, 19, 2240 (1980).
- (38) Electrochemistry of Titanium (IV) in Basic n-Butylpyridinium Chloride and Aluminum Chloride in Presence of Oxide, Hogne Linga, Z. Stojek and R. A. Osteryoung, submitted to *J. Am. Chem. Soc.*
- (39) Electrochemical Studies in Low Temperature Molten Salt Systems Containing Aluminum Chloride, B. J. Welch and R. A. Osteryoung, submitted to *J. Electroanal. Chem.*, July 1980 as publication of special edition of Proceedings of the Fifth Australian Electrochemistry Conference, Perth, August 18-22, 1980.
- (40) Substituent Effects in Iron Diimine Complexes: Correlations with Thermodynamic Properties in a Room Temperature Molten Salt, Helena Li Chum, D. Koran and R. A. Osteryoung, submitted to *Inorg. Chem.*
- (41) Electrochemical and Spectroscopic Studies of 9,10 Anthraquinone in a Room Temperature Molten Salt, Graham T. Cheek and R. A. Osteryoung, to be submitted to *J. Electrochem. Soc.*
- (42) Reverse Pulse Polarography, Janet Osteryoung and Emilia Kirowa-Eisner, *Anal. Chem.*, 52, 62-66 (1980).
- (43) Reverse Pulse Voltammetry: Application to Second Order Following Reactions, Janet Osteryoung, D. Talmor, J. Hermolin, and Emilia Korowa-Eisner, *J. Phys. Chem.*, In Press.
- (44) Reverse Pulse Voltammetry: Application to Coupled Chemical Reactions. Janet Osteryoung, J. Hermolin, D. Talmor, S. Kasliti-Kaplan, and Emilia Kirowa-Eisner, To be submitted to *J. Electroanal. Chem.*
- (45) Spherical Diffusion and Shielding Effects in Reverse Pulse Polarography, Timothy R. Brumleve and Janet Osteryoung to be submitted to *J. Electroanal. Chem.*
- (46) Differential Normal Pulse Voltammetry, Koichi Aoki, R. A. Osteryoung, and Janet Osteryoung, *J. Electroanal. Chem.*, 110, 1-18 (1980).
- (47) Differential Normal Pulse Voltammetry in the Alternative Pulse Mode for Reversible Electrode Reactions, Timothy R. Brumleve, John J. O'Dea, Robert Osteryoung, and Janet Osteryoung, submitted to *Anal. Chem.*
- (48) Differential Normal Pulse Voltammetry for Totally Irreversible Reactions, Timothy R. Brumleve and Janet Osteryoung, to be submitted to *Anal. Chem.*
- (49) Application of Differential Normal Pulse Voltammetry to Totally Irreversible Reactions, Timothy R. Brumleve, R. A. Osteryoung and Janet Osteryoung, To be submitted to *Anal. Chem.*



Meeting Presentation and Seminars--Related to Grant Activity

(\*Invited Presentations)

1971

- \*R. A. Osteryoung, "Computerized Electrochemical Experimentation," Dept. of Chemistry Colloquia, Pennsylvania State University, University Park, PA, May 27, 1971.
- \*L. G. Boxall, H. L. Jones and R. A. Osteryoung, "Electrochemical Studies in Aluminum Chloride Melts," N. W. Regional ACS Meeting, Bozeman, Montana, June, 1971.
- \*R. A. Osteryoung, "Application of a Computer-Based Pulse Polarographic System in Molten Salt Studies," Gordon Research Conference on Molten Salts, Kimball Union Academy, Meriden, N. H., August, 1971.
- \*R. A. Osteryoung, "Use of a Mini-Computer in Electrochemical Studies," Symposium on Mini-Computers in the Research & Teaching Laboratory, American Chemical Society Meeting, Washington, D. C., September, 1971.

1972

- \*R. A. Osteryoung, "Chemistry and Electrochemistry in Fused Salts," Baylor University (ACS Waco Station), May 8, 1972.
- \*R. A. Osteryoung, "Computer Controlled Electrochemical Experimentation," Texas Christian University (Dallas-Ft. Worth ACS Section), May 9, 1972.
- \*R. A. Osteryoung, "Computer Controlled Electrochemical Experimentation," (San Antonio, Texas ACS Section), May 10, 1972.
- \*R. A. Osteryoung, "Computer Controlled Electrochemical Experimentation," Texas A & M (College Station, Texas ACS Section), May 11, 1972.
- \*R. A. Osteryoung, "Computer Controlled Electrochemical Experimentation," Midwestern University (Wichita Falls, Texas/Duncan, Okla. ACS Section), May 12, 1972.
- \*H. L. Jones, L. G. Boxall and R. A. Osteryoung, "Organic Electrochemistry in Aluminum Halide Melts," Rocky Mountain Regional ACS Meeting, Ft. Collins, CO, June 1972.

1973

- \*R. A. Osteryoung, H. L. Jones and L. G. Boxall, "Electrochemical Studies in Molten Chloroaluminates," Symposium on Fused Salt Technology, Electrochemical Society Meeting, Chicago, May 8-13, 1973.

- \*R. A. Osteryoung, R. H. Abel, L. G. Boxall and B. H. Vassos, "An Introduction to the On-Line Use of Digital Computers in Electrochemistry," Plenary Lecture at Symposium on Electrochemical Measurements, by Digital Computer, Electrochemical Society Meeting, Chicago, May 8-13, 1973.

#### 1974

- D. E. Bartak and R. A. Osteryoung, "Oxidation of Tetramethylbenzidine in Chloroaluminate Melts," Electrochemical Society, San Francisco, CA, May, 1974.
- \*R. A. Osteryoung, "Chemistry in Aluminum Chloride Melts," Fifth International Conference in Non-Aqueous Solutions, International Union of Pure and Applied Chemistry, Vienna, July 10-12, 1974.
- \*R. A. Osteryoung, "Electrochemical Studies in Fused Salts," First Latin American Electrochemistry and Corrosion Meeting (ABRACO), Rio de Janeiro, Brazil, Oct. 21-25, 1974.

#### 1975

- \*R. A. Osteryoung, "Some Applications of Pulse Techniques to Analytical Chemistry and Electrochemistry," University of Brussels, Brussels, Belgium, March 17, 1975.
- V. R. Koch, L. L. Miller and R. A. Osteryoung, "Electroinitiated Fiedel-Crafts Transalkylation in a Room Temperature Molten Salt Medium," Mile High Electrochemistry Symposium, Colorado Electrochemical Society Section, Fort Collins, CO, May 3, 1975.
- K. A. Paulsen and R. A. Osteryoung, "Electrochemical Studies of Sulfur in Molten Sodium Tetrachloroaluminate," Fall Meeting, American Chemical Society, Chicago, Illinois, August 1975.
- \*R. A. Osteryoung, "Electrochemical Studies in Molten Sodium Chloride-Aluminum Chloride," Colorado College, Colorado Springs, Colorado, December 4, 1975.

#### 1976

- \*R. A. Osteryoung, "Chemistry and Electrochemistry in Aluminum Chloride Molten Salt Systems," International Symposium on Molten Salts, Spring Meeting, The Electrochemical Society, Washington, D. C., May 2-7, 1976.
- \*R. A. Osteryoung, "Electrochemical Studies in Molten Chloroaluminates," Gould, Inc., Rolling Meadows, Illinois, March 4, 1976.
- \*R. A. Osteryoung, "Chemical and Electrochemical Studies in Molten Chloroaluminate Solvents," Dept. of Chemistry Colloquium, Purdue University, W. Lafayette, Indiana, March 23, 1976.

\* V. R. Koch, L. L. Miller and R. A. Osteryoung, "Electroinitiated Friedel-Crafts Transalkylations in a Room Temperature Molten Salt Medium," Symposium on Electroorganic Oxidations, Electrochemical Society Meeting, Washington, D. C., May 2-7, 1976.

H. Chum, D. Koran and R. A. Osteryoung, "Studies in Room Temperature Molten Salt Systems," Mile High Electrochemistry Second Annual Symposium, Rocky Mountain Section, Electrochemical Society Meeting, Fort Collins, CO, May 15, 1976.

\* K. Paulsen and R. A. Osteryoung, "Electrochemical Studies in Molten Chloroaluminates: Sulfur and Sulfides," Rocky Mountain Regional Meeting, American Chemical Society, Laramie, Wyoming, June 17-19, 1976.

\* H. L. Jones, K. Paulsen, D. Bartak, J. Robinson and R. A. Osteryoung, "Acid-Base Dependent Electrochemistry in Molten Sodium Tetrachloroaluminate Solvents," Symposium on Spectroscopic and Electrochemical Characterization of Solute Species in Non-Aqueous Solvents, American Chemical Society Meeting, August 29-September 3, 1976.

Helena Li Chum, D. Koran and R. A. Osteryoung, "Room Temperature Molten Salts and Its Mixtures with Benzene - New Solvents for Spectroscopic and Electrochemical Studies," Physical Chemistry Division, American Chemical Society Meeting, San Francisco, CA, August 29-September 3, 1976.

\* R. A. Osteryoung, "Chemistry & Electrochemistry in Molten Chloroaluminates (Including Some Possible Applications to Sulfide Ores)," Environmental Impact Center, Newton, MA, October 27, 1976.

#### 1977

J. Robinson and R. A. Osteryoung, "The Electrochemical Behavior of Selenium in  $\text{NaCl-AlCl}_3$  Melts," American Chemical Society Meeting, New Orleans, LA, March 20-25, 1977.

\* R. A. Osteryoung, "Chemical and Electrochemical Studies in Molten Chloroaluminates," Department of Chemistry, University of Chicago, Chicago, IL, February 7, 1977.

\* R. A. Osteryoung, "Electrochemical Studies in Molten Chloroaluminates," 4th Biennial Air Force Electrochemistry Conference, F. J. Seiler Research Laboratory, U. S. Air Force Academy, CO, April 28-29, 1977.

\* R. A. Osteryoung, "Recent Trends in Electrochemical Research," Environmental Research and Development Administration, Materials Science Workshop in "Electrochemistry and Thermodynamics," Argonne National Laboratory, Argonne, IL, May 19-20, 1977.

\* R. A. Osteryoung, "Acid-Base Dependent Electrochemistry in Aluminum Halide Molten Salts," Gordon Research Conference on Molten Salts and Metals, Tilton, N. H., July 24-29, 1977.

\*R. A. Osteryoung, "Chemistry and Electrochemistry in Molten Chloroaluminates," Georgia Institute of Technology, Atlanta, GA, October 12, 1977.

\*J. Robinson and R. A. Osteryoung, "The Electrochemistry of Selenium in Molten Sodium Tetrachloroaluminates," 3rd Annual Mile High Symposium on Electrochemistry, Rocky Mountain Section of the Electrochemical Society, Fort Collins, CO, May 7, 1977.

#### 1978

\*R. A. Osteryoung, "Chemistry and Electrochemistry in Molten Chloroaluminates," Georgetown University, Washington, D. C., February 9, 1978.

R. A. Osteryoung and B. Gilbert, "Electrochemistry of Nickel in Molten Sodium Tetrachloroaluminates," National American Chemical Society Meeting, Anaheim, CA, March 12-17, 1978.

\*R. A. Osteryoung, "Chemistry and Electrochemistry in Molten Chloroaluminates," Wichita State University, Wichita, KS (April, 1978).

\*R. A. Osteryoung, "Electrochemistry in Molten Chloroaluminates," Naval Surface Weapons Center, White Oak, Maryland (May, 1978).

\*R. A. Osteryoung, "Acid Base Dependent Electrochemistry In Chloroaluminate Melts," Conference on Highly Concentrated Aqueous Solutions and Molten Salts, Oxford University, Oxford, England (July 5-7, 1978).

\*R. A. Osteryoung, "Acid Base Dependent Chemistry and Electrochemistry in Chloroaluminate Melts," The University of Southampton, Southampton, England (July 10, 1978).

\*R. A. Osteryoung, R. J. Gale, J. Robinson, R. Bugle and B. Gilbert, "Electrochemical Studies in a Room Temperature Molten Salt," Second International Meeting on Molten Salts, The Electrochemical Society, Pittsburgh, Pennsylvania (October 15-20, 1978).

\*R. A. Osteryoung, "Acid-Base Dependent Chemistry and Electrochemistry in Molten Chloroaluminates," Gould, Inc., Rolling Meadows, IL, November 16, 1978.

\*R. A. Osteryoung, "Acid-Base Dependent Chemistry and Electrochemistry in Molten Chloroaluminates," Colorado Section, American Chemical Society, University of Colorado, December 12, 1978 (Colorado Section Award Talk).

#### 1979

\*R. A. Osteryoung, "Room Temperature Molten Salts: A New Class of Solvents," Gordon Research Conf. on Electrochemistry, Santa Barbara, CA, Jan. 7-12, 1979.

\*R. A. Osteryoung, "Studies in Molten Chloroaluminates," Oak Ridge National Laboratory, Oak Ridge, TN, Feb. 2, 1979.

1980

- \*R. A. Osteryoung, "Chemistry and Electrochemistry in Molten Chloroaluminates," Canisius College, Buffalo, N. Y., Feb. 14, 1980.
- \*R. A. Osteryoung, "Chemical and Electrochemical Studies in Molten Chloroaluminates," Colloquium, Department of Chemistry, University of Pittsburgh, Pittsburgh, PA, Feb. 28, 1980.
- \*R. A. Osteryoung, "Acid-Base Dependent Chemistry and Electrochemistry in Molten Chloroaluminates," University of North Dakota, Grand Forks, North Dakota (Department Colloquia), March 14, 1980.
- \*R. A. Osteryoung, "Electrochemical Studies in Molten Chloroaluminates," Sioux Valley American Chemical Society Section, Sioux Falls, South Dakota, March 15, 1980 (ACS Tour Speaker).
- \*R. A. Osteryoung, "Acid-Base Dependent Chemistry and Electrochemistry in Chloroaluminate Melts, Pittsburgh Section, The Electrochemical Society, Spring Meeting, May 23, 1980.
- \*R. A. Osteryoung, Graham Cheek and Hagne Linga, "Studies in Room Temperature Molten Salts," Third International Symposium on Molten Salts, The Electrochemical Society, Fall Meeting, Hollywood, Florida, October 5-10 (1980).
- \*R. A. Osteryoung, "Electrochemical Studies in Molten Chloroaluminates," Sioux Valley American Chemical Society Section, Sioux Falls, South Dakota, March 15, 1980 (ACS Tour Speaker).
- \*R. A. Osteryoung, "Acid-Base Dependent Chemistry and Electrochemistry in Chloroaluminate Melts, Pittsburgh Section, The Electrochemical Society, Spring Meeting, May 23, 1980.
- \*R. A. Osteryoung, Graham Cheek and Hagne Linga, "Studies in Room Temperature Molten Salts," Third International Symposium on Molten Salts, The Electrochemical Society, Fall Meeting, Hollywood, Florida, October 5-10 (1980).
- \*Janet Osteryoung, "Recent Advances in Pulse Voltammetry," Pittsburgh Conference, Atlantic City, March, 1980.
- Janet Osteryoung and Edmund Hurdle, Jr, "Construction of a Potentiostat for Use in Computer-Assisted Electrochemistry", 34th Eastern Colleges Science Conference, Cortland, N.Y., April, 1980.
- \*Janet Osteryoung, "The Use of Pulse Techniques for Detection of Intermediate Products of Electrode Reactions," J. Heyrovsky Memorial Congress on Polarography, Prague, Czechoslovakia, August, 1980.
- \*Janet Osteryoung, "Recent Advances in Pulse Voltammetry", 3rd Brno Symposium on Molecular Biophysics: Electroanalysis of Biopolymers, Kupařovice Castle, Czechoslovakia, August-September, 1980.

\*R. A. Osteryoung, J. Robinson and R. J. Gale, "Oxidation of Aromatic Hydrocarbons in a Room Temperature Molten Salt," Symposium on Electrochemistry and Spectroscopy in Melts, American Chemical Society/Chemical Society of Japan Congress, Honolulu, Hawaii, April 1-6, 1979.

James Robinson and R. A. Osteryoung, "Electrochemical Studies on Some Aromatic Amines in a Room Temperature Molten Salt," Spring Meeting, The Electrochemical Society, St. Louis, MO, May 11-16, 1979.

\*R. J. Gale, A. J. Nozik and R. A. Osteryoung, "Electrochemical Characterization of the Semiconductor  $\text{TiO}_2$ -Room Temperatures Molten Salt Interface," Rocky Mt. Regional Electrochemical Society Meeting, Ft. Collins, CO (June, 1979).

\*G. Cheek and R. A. Osteryoung, "Electrochemical and Infrared Studies of Quinones in a Room Temperature Molten Salt," Gordon Research Conference on Molten Salt," Gordon Research Conference on Molten Salts and Metals, Brewster Academy, Wolfeboro, New Hampshire (August 20-24, 1979).

\*R. J. Gale, J. Robinson and R. A. Osteryoung, "Acid Base Dependent Chemistry and Electrochemistry in Room Temperature Chloroaluminate Melts," Plenary Lecture, 30th Meeting, International Society of Electrochemistry, Trondheim, Norway (August 26-31, 1979).

R. A. Osteryoung, G. Cheek and R. J. Gale, "Correlation of Studies in Quinones in a Room Temperature Molten Salt," Symposium, American Chemical Society, Washington, D. C. (September 9-14, 1979).

G. Cheek and R. A. Osteryoung, "Electrochemical and Spectroscopic Studies in Quinones in a Room Temperature Molten Salt," Northeast Regional American Chemical Society Meeting, Syracuse, N. Y. (Oct. 2-5, 1979).

\*R. A. Osteryoung, "Simple Chemistry in Molten Salts," State University College at Brockport, Brockport, N. Y., Oct. 17, 1979.

\*R. A. Osteryoung, "Chemical and Electrochemical Studies in Molten Chloroaluminates," Colloquium, Department of Chemistry, State University College at Buffalo, Buffalo, N. Y., Nov. 7, 1979.

Janet Osteryoung and T. R. Brumleve, Reverse Pulse Polarography: Spherical Diffusion and Depletion Effects, 178th ACA National Meeting, Washington, D. C., September, 1979.

Janet Osteryoung, Koichi Aoki and R. A. Osteryoung, "Modified Differential Pulse Voltammetry: Theory", 9th NE Regional ACS Meeting, Syracuse, October, 1979.

\*Janet Osteryoung, Recent Advances in Pulse Polarography, Georgetown University, Washington, D. C., November, 1979